



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<p>(54) Title: <b>METHOD OF ENCAPSULATING FOOD OR FLAVOR PARTICLES USING WARM WATER FISH GELATIN, AND CAPSULES PRODUCED THEREFROM</b></p> <p><b>(57) Abstract</b></p> <p>This invention is directed to a method of forming microencapsulated food or flavor capsules as well as the capsules produced by the method. The method includes providing food or flavor particles to be encapsulated, and forming a mixture of a warm water fish gelatin and the food or flavor particles in aqueous media. The method further includes microencapsulating the particles with the gelatin at elevated temperatures by complex coacervation to form the microencapsulated capsules. If desired, the method may further include the step of separating the capsules. In a preferred form, the method is conducted at a temperature of about 33 °C to about 35 °C. Preferably, the warm water fish gelatin used in the encapsulation method has a bloom of from about 150 to about 300 bloom, more preferably from about 250 to about 300 bloom. Many different kinds of food or flavor particles may be used, such as for example, vegetable oil, lemon oil, garlic flavor, apple flavor or black pepper. The invention also is directed to the food or flavor capsules produced by the method.</p>			

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METHOD OF ENCAPSULATING FOOD OR FLAVOR PARTICLES  
USING WARM WATER FISH GELATIN, AND CAPSULES  
PRODUCED THEREFROM

Background of the Invention

This invention relates to microencapsulation of food or flavor particles, and more particularly to microencapsulation of food or flavor particles by complex coacervation at elevated temperatures using a warm water fish gelatin.

U.S. Patent No. 5,035,896 to Apfel et al. teaches water insoluble drugs coated by simple coacervation using a low bloom fish gelatin which coacervates at ambient temperature, i.e., room temperature of about 16°-27°C. However, the '896 patent does not disclose the use of fish gelatin in encapsulating other kinds of particles. Furthermore, the '896 coacervation process is limited to ambient or room temperature coacervation.

Summary of the Invention

This invention is directed to a method of forming

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microencapsulated food or flavor capsules as well as the capsules produced by the method. The method includes providing food or flavor particles to be encapsulated, and forming a mixture of a warm water fish gelatin and the food or flavor particles in aqueous media.

5       The method further includes microencapsulating the particles with the gelatin at elevated temperatures by complex coacervation to form the microencapsulated capsules. If desired, the method may further include the step of separating the capsules.

10      In a preferred form, the method is conducted at a temperature of about 33°C to about 35°C. Preferably, the warm water fish gelatin used in the encapsulation method has a bloom of from about 150 to about 300 bloom, more preferably from about 250 to about 300 bloom. Many different kinds of food or flavor particles may be used, such as for example, vegetable oil, lemon oil, 15     garlic flavor, apple flavor or black pepper. The invention also is directed to the food or flavor capsules produced by the method.

15      This invention offers several benefits and advantages. For example, the method provides a way of encapsulating food or flavor particles using fish gelatin instead of other gelatin such as 20     bovine or porcine gelatin. Also, the fish gelatin employed is a kosher fish gelatin; therefore, any kosher food or flavor particle will remain kosher upon encapsulation. In addition, the coacervation method is conducted at elevated temperatures, and does not require ambient/room temperature for coacervation.

Detailed Description of the Invention

The invention is directed to a method of forming microencapsulated food or flavor capsules, as well as to the capsules produced from the method. The method includes providing food or flavor particles to be encapsulated, and forming a mixture of a warm water fish gelatin and the food or flavor particles in an aqueous media. The method further includes microencapsulating the particles with the gelatin at elevated temperatures by complex coacervation to form the microencapsulated capsules.

The warm water fish gelatin used in the method may be any warm water fish gelatin. However, in a preferred form of the invention, the fish gelatin has a bloom of from about 150 to about 300 bloom, and more preferably, a bloom of from about 250 to about 300 bloom. Such high bloom warm water fish gelatins are available from Food Industries Testing of Miami, Florida under the product name Gelcine KFX. One of the advantages of using the fish gelatin is that it is kosher. Therefore, kosher food or flavor particles retain their kosher status upon encapsulation with the fish gelatin.

As stated above, the method further includes microencapsulating the particles with the gelatin at elevated temperatures by complex coacervation. As used herein, "elevated temperatures" means temperatures above ambient or room temperatures, i.e., above 16°-27°C. During complex coacervation, a protein coating is formed over the food or flavor particles to produce

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microencapsulated capsules. The protein coating may then be cross linked by covalent or ionic bonding with a cross linking agent, and if desired, the capsules may be spray dried or otherwise dewatered at a suitable temperature for the removal of water.

5                 By employing the method of this invention, food or flavor particles in amounts of up to 95% by weight, typically within the range of about 50% to 95% by weight, are encapsulated in a protein coating, i.e., at a ratio of about 10:1 to about 5:1 of particles to coating. Usually, about 70% to about 95% by weight  
10                 particle encapsulation is achieved. In another feature of the invention, a drying aid is added to the coacervated emulsion of food or flavor particles before spray drying or dewatering. This drying aid has the effect of lubricating these spray dried, or otherwise dewatered particles and provides for uniform distribution of the  
15                 particles.

A variety of food or flavor particles may be encapsulated, such as for example, vegetable oil, lemon oil, garlic flavor, apple flavor or black pepper. Examples of other flavor oils include flavoring aromatic compounds and/or oils, oleoresins and  
20                 extracts derived from plants, leaves, flowers, fruits and so forth, and combinations thereof. Flavor oils include cinnamon oil, oil of wintergreen, peppermint oil, bay oil, thyme oil, spearmint oil, cassia oil, and the like. Artificial, natural and synthetic fruit flavors such as vanilla, and citrus oils including lemon, orange, grapefruit, lime, and

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fruit essences including apple, pear, peach, strawberry, cherry, and so forth, may be used. These flavoring oils may be used individually or in a mixture as is well known in the art.

The coating layer, as prepared by complex coacervation comprises colloidal materials which must be hydrophilic, gellable and ionizable. Colloidal materials may be selected from the group consisting of a warm water fish gelatin, alginates, casein, gum arabic, carboxymethylcellulose and the like, and mixtures thereof.

Complex coacervation is practiced usually by first forming a solution of a first colloidal material, such as the warm water fish gelatin, in water above its gelation temperature.

Separately, a second colloidal material, such as gum arabic or carboxymethylcellulose for example, is added to water to form a clear solution. The two solutions then are mixed, the temperature may be reduced, and the flavor oil is mixed into the resulting solution at a mixing speed to form the desired emulsion. Complex

coacervation, or aggregation to uniformly distribute the colloidal materials around the food or flavor particles, then is carried out by diluting the emulsion with water, or adjusting the pH and allowing an elapsed time for the colloid to coat the particles. Then, in order to render the gelatin capsules insoluble, it is usually necessary to cross link the colloidal coating on the particles, and in a preferred form,

glutaraldehyde is used to cross link the gelatin coating surrounding the particles. Alum also may be used to cross link the coated food

Example 2**Spray-Dried, Free-Flowing Capsules of Flavor Particles And Cross-Linked Warm Water Fish Gelatin Coating**

The aqueous suspensions of lemon oil, garlic flavor and  
5 apple flavor capsules prepared in Example 1 above were further  
processed to form dry powders of flavor capsules. In order to form  
the dry powders, silicon dioxide was mixed into the slurries or  
aqueous suspensions to act as a drying aid upon spray-drying. The  
suspensions were then spray-dried using the spray-drying technique  
10 discussed above. This process resulted in dry, free flowing  
microcapsules.

## I CLAIM:

1. A method of forming microencapsulated food or flavor capsules comprising the steps of:

providing food or flavor particles to be encapsulated;

5 forming a mixture of a warm water fish gelatin and said food or flavor particles in aqueous media; and

microencapsulating said particles with said gelatin at elevated temperatures by complex coacervation to form said microencapsulated capsules.

10 2. The method of claim 1 conducted at a temperature of about 33°C to about 35°C.

3. The method of claim 1 further comprising the step of separating said capsules.

15 4. The method of claim 1 wherein said fish gelatin has a bloom of from about 150 to about 300 bloom.

5. The method of claim 3 wherein said fish gelatin has a bloom of from about 250 to about 300 bloom.

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6. The method of claim 1 wherein said particles to be encapsulated are selected from the group consisting of vegetable oil, lemon oil, garlic flavor, apple flavor and black pepper.

7. The product produced by the method of claim 1.

5 8. The product produced by the method of claim 2.

9. The product produced by the method of claim 3.

10. The product produced by the method of claim 6.

11. The product produced by the method of claim 1  
wherein said product is kosher.

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12. A method of forming microencapsulated liquid flavor capsules comprising the steps of:

providing liquid flavor particles to be encapsulated;

forming a mixture of a warm water fish gelatin and said

5 liquid flavor particles in aqueous media; and

microencapsulating said particles with said gelatin at elevated temperatures by complex coacervation to form said microencapsulated capsules.

## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 95/16628

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 A23P1/04 A23L1/22

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 A23P A23L A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO,A,93 19621 (TASTEMAKER) 14 October 1993 see the whole document ---	1-11
Y	EP,A,0 347 751 (F.HOFFMAN-LA ROCHE) 27 December 1989 see column 1, line 32 - column 3, line 20 see examples 1,3,5-9 ---	1-11
Y	EP,A,0 273 823 (MERO ROUSSELOT SATIA) 6 July 1988 see the whole document ---	1-11
Y	EP,A,0 346 879 (WARNER-LAMBERT) 20 December 1989 see the whole document & US,A,5 035 896 cited in the application ---	1-11
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
12 April 1996	26.04.1996

## Name and mailing address of the ISA

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## INTERNATIONAL SEARCH REPORT

PCT/US 95/16628  
International Application No

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	P.B.DEASY: "Microencapsulation and Related Drug Process" 1984 , M.DEKKER , NEW YORK - USA XP002000439 pp.64-75 see page 69 ---- WO,A,91 17821 (DANOCHEMO) 28 November 1991 see the whole document -----	2
		1

## INTERNATIONAL SEARCH REPORT

Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)		Publication date
W0-A-9319621	14-10-93	AT-T-	129855	15-11-95
		AU-B-	3969893	08-11-93
		CA-A-	2130442	14-10-93
		CZ-A-	9402365	17-05-95
		DE-D-	69300768	14-12-95
		EP-A-	0633732	18-01-95
		FI-A-	944517	29-09-94
		JP-T-	8501441	20-02-96
		SK-A-	114994	05-01-95
EP-A-347751	27-12-89	AU-B-	623908	28-05-92
		AU-B-	3657389	04-01-90
		HK-A-	142595	15-09-95
		IE-B-	62542	08-02-95
		JP-A-	2051594	21-02-90
		NO-B-	177374	29-05-95
		PT-B-	90948	30-11-94
		US-A-	5478569	26-12-95
EP-A-273823	06-07-88	FR-A-	2608456	24-06-88
		CA-A-	1323307	19-10-93
		JP-A-	63197540	16-08-88
		US-A-	5051304	24-09-91
EP-A-346879	20-12-89	US-A-	5035896	30-07-91
W0-A-9117821	28-11-91	AU-B-	7903191	10-12-91
		EP-A-	0530277	10-03-93